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(54) Title: RECORDING TRANSPARENCY AND METHOD

(57) Abstract

A recording transparency and method of preparing the same from water solution are disclosed. The transparency is receptive to a wide variety of inks and other indicia, exhibits rapid ink drying times, excellent dot size and shape retention, and excellent water resistance and stability. The transparency comprises a transparent substrate coated with one or a mixture of hydroxyethyl cellulose polymers from aqueous solution.

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# RECORDING TRANSPARENCY AND METHOD

## BACKGROUND OF THE INVENTION

### Field of the Invention

This invention relates generally to recording or printing media and, more particularly, this invention relates to a recording transparency useful in connection with ink jet and other recording processes.

### Description of Related Art

The use of recording transparencies in connection with, for example, overhead projection systems is well know. Typical recording transparencies comprise a flexible substantially transparent film usually of a hydrophobic polymeric material such as polyester, with a surface coating which accepts various indicia, such as inks from ink jet printers or pens and the like.

It is desirable that the surface coating be substantially clear and capable of providing high density images which are permanent and smear resistant. It is also important that images imparted to the coating dry quickly. The latter characteristic is generally a function of the rate of ink sorption, while smear resistance is principally determined by the hydrolytic stability of the coating.

While some prior coatings used in recording transparencies have provided acceptable ink drying times and/or smear resistance for various specific types of inks, it is desirable for a recording transparency to accept any of a wide range of indicia, including indicia from ink jet printers, solvent base inks such as used in marker pens, and xerographic toners. Such transparencies would be highly versatile in a commercial setting, allowing users to utilize more than one type of printer, marker or copier to produce a given

transparency. Heretofore, recording transparencies having the versatility to accept different types of inks and/or toners while providing fast drying times and smear resistance have not been known.

Also, because of the hydrophobic nature of the film surface, the coating of the film with the ink receptive layer is usually done from a solvent. It is a major departure from the art that the coating of clear film in this invention is done from aqueous solution or suspension. Coating from water is not only safer but also avoids environmental problems with the storage and recovery or disposal of the solvents.

## SUMMARY OF THE INVENTION

It is an object of the present invention to overcome one or more of the problems described above.

According to the present invention, a recording transparency comprising a substantially transparent substrate having a hydrophilic surface bearing a substantially transparent coating is provided. The coating is applied to the hydrophilic surface of the substrate by application of an aqueous coating solution, followed by evaporation of the water in the solution.

The coating solution comprises an aqueous solution of at least one hydroxyethyl cellulose polymer in a sufficiently high concentration to provide a desirably high rate of indicia sorption and drying, while having a sufficiently high viscosity to be readily coatable on the substrate surface. The coating solution optionally may contain a surfactant to promote leveling and adhesion to the surface, as well as hydrated alumina in order to impart pencil tooth to the surface if desired.

Other objects and advantages of the invention will be apparent to those skilled in the art from a review of the following detailed description, taken in conjunction with the appended claims.

## DETAILED DESCRIPTION OF THE INVENTION

According to the present invention, a substantially clear transparency coating having the attributes of fast drying time (i.e. rapid sorption of inks or other indicia applied to the coating) as well as excellent hydrolytic stability (providing smear resistance) and which is readily receptive to a wide variety of indicia (including water and solvent based inks, ink jet inks, dry xerographic toners, pencil indicia, etc.) is provided for the first time. These properties result from the formation of a transparency coating by the application and drying of an aqueous coating solution of one or more hydroxyethyl cellulose polymers. The polymer is present in a concentration and has an average molecular weight and molecular weight distribution such that there is sufficient relatively low molecular weight polymer to provide good ink sorption rate characteristics, with sufficient relatively high molecular weight polymer to impart a solution viscosity which allows ready coating of the substrate.

Thus, it has been found that coating characteristics balancing a desirably high rate of ink sorption with the desirably high hydrolytic stability provided by higher molecular weight materials can be provided using hydroxyethyl cellulose polymers either singly or in admixture.

More specifically, it has been found that an aqueous solution containing one or more hydroxyethyl cellulose polymers in a total concentration range of about 0.25 to about 7 wt. percent, inclusive, with a distribution of polymer molecular weight to provide a solution viscosity of between about 100 and about 4000 cps, inclusive (as measured at 25°C), will provide a transparency coating having all of the desirable properties set forth above.

Preferably, the coating solution in a concentration within the range of about 2 to 6 wt. percent, inclusive, and the solution viscosity is in the range of about 200 to about 2000 cps, inclusive. The solution viscosity is highly preferably in the range of about 1000 to 2000 cps, inclusive. (Reference herein to solution viscosity is understood to mean LVF Brookfield viscosity as measured at 25°C.)

The substantially transparent substrate will generally be of a polymeric material, which typically will be hydrophobic. It is necessary only that the surface to be coated by hydrophilic, and techniques for treating hydrophobic materials to render their surfaces hydrophilic are well known in the art.

A preferred transparency substrate material is a polyester film marketed by Imperial Chemical Industries (ICI) under the trademark MELINEX, Type 519 or 582. This polyester film is hydrophobic, but is pretreated to render its surfaces hydrophilic. Another transparency substrate material which is believed to be useful is market by Hoechst under the trademark HOSTAPHAN 4660.

Hydroxyethyl cellulose (HEC) polymers are commercially available. Those which are currently preferred are marketed by Union Carbide Corporation under the trademark CELLOSIZE. CELLOSIZE hydroxyethyl cellulose is available in a wide range of viscosity grades which are believed to differ primarily in average molecular weight and molecular weight distribution.

Although the actual average molecular weight and molecular weight distributions of the Union Carbide CELLOSIZE HEC products are not believed to be publicly available, viscosity data for these materials, which are directly correlatable to molecular weight, are available.

According to the invention, it has been found that the use in a coating solution of relatively high

molecular weight hydroxyethyl cellulose polymers provides good water resistivity and stability to transparency coatings. However, the use of only high molecular weight materials tends to provide very viscous solutions and, because of relatively low water solubilities of HEC polymers provides coatings which have a poor capacity for ink jet inks, for example.

Conversely, the use of low molecular weight HEC polymers provides a solution having a viscosity which allows ready coating, but which is relatively sensitive to relative humidity and water, thus providing poor smear resistance.

Thus, it has been found that the use in aqueous solution of a single hydroxyethyl cellulose polymer having an intermediate average molecular weight or a mixture of relatively low and high molecular weight materials in aqueous solution provides an excellent balance between sorption capacity and drying rate and on the one hand and hydrolytic stability on the other. It is believed that the ability to provide quick drying times with excellent image formation is a result of having a sufficiently high number of low molecular weight molecules in the coating, while hydrolytic stability results from the presence of a sufficiently high number of high molecular weight molecules in the coating.

It has been further found that these desirable properties are correlatable with the polymer concentration and the viscosity of the coating solution.

Thus, according to the invention, an aqueous solution comprising a sorbent consisting essentially of one or more hydroxyethyl cellulose polymers having a total polymer concentration in the range of about 0.25 to about 7 wt. percent, inclusive, as well as a solution viscosity of between about 100 and 4000 cps, inclusive, will provide good results.

Preferably, the polymer concentration is in the range of about 2 to 6 wt. percent, inclusive, highly preferably in the range of about 3 to 4 wt. percent, and the viscosity is in the range of about 200 to about 2000 cps, highly preferably in the range of about 1000 to 2000 cps, inclusive. Conveniently, the solution may comprise a mixture of hydroxyethyl cellulose polymers dissolved in water.

If a single hydroxyethyl cellulose polymer is to be used, a convenient choice is Union Carbide's CELLOSIZE QP40, which is characterized as having a 2 wt. percent aqueous solution viscosity in the range of 80-145 cps at 25°C using a number 1 spindle at 30 rpm.

If a mixture of two or more HEC polymers is to be used, a convenient low molecular weight polymer is CELLOSIZE QP09H or QP09L. Grade QP09L has a viscosity range of 75-112 cps (5 wt. percent aqueous solution, spindle number 1, 30 rpm) and grade QP09H has a viscosity range at 25°C of 113-150 cps (5 wt. percent aqueous solution, spindle number 1, 30 rpm).

Although CELLOSIZE QP09H or L grade hydroxyethyl cellulose may be used alone to provide good ink sorption characteristics, they are preferably used in admixture with at least one relatively high molecular weight HEC, such as the preferred QP4400 or the intermediate molecular weight QP300. QP4400 HEC has a Brookfield viscosity at 25°C of 4800 to 5600 cps (2 wt. percent aqueous solution, spindle number 4, 60 rpm) while QP300 HEC has a viscosity range of 250-400 cps at 25°C (2 wt. percent aqueous solution, spindle number 2, 60 rpm).

Aqueous solutions of QP09 with QP4400 or with a mixture of QP4400 and QP300 give clear transparency coatings with sufficient ink sorption capacity for the Hewlett Packard (HP), Siemens, and Canon ink jet printers. Drying times for the HP unit (the slowest drying of these units) are on the order of 8-25 seconds

to a nonsmudging state using a solution containing 6 wt. percent QP09H, and 1 wt. percent of a mixture of QP4400 and QP300. This type of coating is also receptive to a glycol based ink in pens which will dry smudge free in less than one second, or a water based ink in pens which will dry to a smudge free state in less than 5 seconds.

The solution preferably should contain no more than about 1.5 wt. percent, preferably 0.3 to 1.5 wt. percent, of the relatively high molecular weight HEC polymer, and this solution should contain no more than 7 wt. percent of all HEC polymers.

If desired, an antifoam agent such as Antifoam T (Andrews) or Nopco's Foam Master DF160L may be used. Although not generally necessary, these materials do not adversely affect performance. Typical usage rates of antifoam materials are about 0.01 wt. percent.

In many cases it will be desirable to utilize a surfactant to promote adhesion of the transparency coating to the substrate. A preferred surfactant is marketed by International Products Corporation under the trademark MICRO. This is a mixed anionic and nonionic surfactant, and it has been found to both promote adhesion and to eliminate occasional surface defects, and does not retard the rate of ink drying. A surfactant concentration range of about 0.05 to 0.2 percent should be used. At higher concentrations, the solution becomes foamy, and below the lower end of the range the effects of the surfactant are insufficient for practical purposes.

In practice, when the transparency is produced by rod coating, a relatively dilute solution and larger rod sizes are preferred. For example, a solution having a 6 wt. percent QP09 concentration is too viscous for rod coating. Also, since QP300 HEC contributes to viscosity with only a marginal benefit to drying rate, it may be eliminated if desired.

Although relatively dilute solutions coated in thicker layers should give very similar results to relatively viscous solutions coated in thin layers, in practice relatively viscous solutions apply more material so that for rod coating a viscosity range of up to 400 poise is desirable, with an optimum near 300 or 400.

The hydrophilic coatings made according to the invention sorb ink from HP, Canon and Siemens ink jet systems with excellent retention of dot shape and size. Also, these coatings sorb ink from glycol based marker pens and water based ink pens, and provide excellent images with xerographic dry toners, including those used in laser printing. They serve extremely well for projection films.

If it is desired to allow the use of pencils on the transparency, a gritty coating which provides good pencil tooth may be obtained by the inclusion in the coating solution of hydrated alumina, preferably with very minor amounts of coated silica. A preferred alumina is marketed by Degussa under the trademark ALUMINA-C, a high surface area (e.g. 100 $m^2/g$) fumed alumina having low average particle size (about 200 nm) so that scattering of light is minimized and the coatings remain relatively clear. Any silica used in the coating should be very fine grained and wax coated in order to minimize scattering. A preferred silica is SCM's Silcron G-530. The coating preparation should be carried out in order to provide as uniform a dispersion of the alumina and silica as possible. To this end, the alumina and silica may be predispersed in water and added to the bulk of the coating solution.

The following table provides examples of various formulations, solution viscosity and ink drying times using the Hewlett Packard ink jet printer:

TABLE

| Sample | QP 09 % | QP 4400 % | QP 300 % | Micro % | Coating Solution Viscosity (cps) | HP Ink Drying Time (Sec) |
|---|---|---|---|---|---|---|
| 1 | 6* | 1.5 | 1.0 | 0.1 | 3800 | 9 |
| 2 | 6 | 1.5 | 1.0 | 0.1 | 3100 | 12 |
| 3 | 6 | 1.5 | --- | 0.1 | 1500 | 13 |
| 4 | 4 | 1 | --- | 0.1 | 380 | 13 |
| 5 | 4* | 1 | --- | 0.1 | 420 | 14 |
| 6 | 3.3* | 1 | 1 | 0.1 | 700 | 12 |
| 7 | 3.3 | 1 | --- | 0.1 | 240 | 17 |
| 8 | 4 | 0.8 | --- | 0.1 | 220 | 16 |

*denotes QP09H; other samples used QP09L

The following exemplary formulations and procedures further illustrate the practice of the invention. 6.5 g QP 09L HEC polymers and 0.2 wt.% Micro surfactant were added to 100 g water and dissolved by stirring. To this solution was added a slurry of 0.8 g Alumina-C hydrated alumina and 0.1 g Silcron G-530 coated silica in 10 to 30 ml water. The resultant slurry was stirred for several hours and then rod coated on a polyester film (Melinex 519) using a size 40 - 55 rod. The resultant coating, after drying, was clear in appearance, gave drying times of 60 and 20 sec with the HP and Canon ink jet printers, accepted both water based and glycol based ink pens and ball point pens (drying times less than 3 sec) and would also accept 5H pencil, K-4 plastic pencil, and normal ball point ink. The pencil lines could be erased, and redrawn in the erased areas.

Other materials tested and found satisfactory included QP 09H HEC at 0.25 - 7% either alone or mixed with 0.3 to 0.75% QP 4400 HEC (total HEC concentration

no more than 7%); Micro surfactant concentrations of 0.05 to 0.2%; Alumina-C alumina levels of 0.75 to 2%; and Silcron G-530 coated silica levels of 0.1 to 0.3%.

The fastest drying of HP Thinkjet ink was observed with 2.5% QP 09H HEC and 0.7% QP 4400 HEC and a fine grained silica. However, the sample was very hazy and opaque. Using 2.5% QP 09H HEC, 0.75% QP 4400 HEC, 1.0% Alumina-C alumina and 0.2% Silcron G-530 silica, drying times of about 4 sec could be obtained.

Preferably, the coating solution contains dimethylolurea (DMU) as a cross-linking agent, in a concentration of about 0.05 wt.% to about 0.4 wt.%, based on the weight of hydroxyethyl cellulose polymer. The optimum concentration is about 0.2 wt.%. Concentrations less than 0.05 wt.% have little effect, and concentrations greater than 0.4 wt.% affect ink sorption.

The use of DMU as set forth above results in cross-linking of the HEC polymers to harden the surface to eliminate blocking problems. Also, the surface is rendered less sensitive to changes in ambient relative humidity.

The foregoing detailed description is given for clearness of understanding only, and no unnecessary limitation should be understood or inferred therefrom, as obvious modifications within the scope of the invention will be apparent to those skilled in the art.

I claim:

1. A recording transparency comprising:

(a) a substantially transparent substrate having a hydrophilic surface; and,

(b) a substantially clear hydrophilic coating applied to said surface by coating said surface with an aqueous coating solution followed by evaporation of the water in said solution, said solution comprising one or more hydroxyethyl cellulose polymers dissolved in water, the concentration, average molecular weight, and molecular weight distribution of said polymer being selected such that said coating solution has a viscosity so as to be readily coatable on said surface and said coating provides a desired high rate of sorption and drying of indicia applied thereto.

2. The transparency of claim 1 wherein the concentration of said polymer in said coating solution is within the range of about 0.25 to 7 wt. percent, inclusive, and the viscosity of said solution is between about 100 and 4000 cps, inclusive, as measured at 25°C.

3. The transparency of claim 2 wherein said concentration is in the range of about 2 to 6 wt. percent, inclusive, and said viscosity is in the range of about 200 to about 2000 cps, inclusive.

4. The transparency of claim 2 wherein said solution comprises a mixture of hydroxyethyl cellulose polymers.

5. The transparency of claim 2 wherein a first said polymer is present in said coating solution and has a relatively low average molecular weight such that a 5 wt. percent aqueous reference solution thereof has a viscosity in the range of about 75 to 150 cps at 25°C.

6. The transparency of claim 5 wherein a second said polymer is present in said solution and has a relatively high average molecular weight such that a 2 wt. percent aqueous reference solution thereof has a viscosity in the range of about 4800 to 5600 cps at 25°C.

7. The transparency of claim 5 wherein a third said polymer is present in said solution and has a relatively high average molecular weight such that a 2 wt. percent aqueous reference solution thereof has a viscosity in the range of about 250 to 400 cps at 25°C.

8. The transparency of claim 6 wherein a third said polymer is present in said solution and has a relatively high average molecular weight such that a 2 wt. percent aqueous reference solution thereof has a viscosity in the range of about 250 to 400 cps at 25°C.

9. The transparency of claim 6, 7 or 8 wherein the concentration in said coating solution of said first polymer is in the range of about 0.25 to 7 wt. percent, inclusive, the concentration of said second polymer is up to about 1.5 wt. percent, and the concentration of said third polymer is in the range of 0 to 1 wt. percent, inclusive, provided however that the total concentration of said polymers is in the range of about 0.25 to 7 wt. percent, inclusive.

10. The transparency of claim 9 wherein said first polymer concentration is in the range of about 2 to 6 wt. percent, inclusive.

11. The transparency of claim 10 wherein said second polymer concentration is about 0.3 to 1.5 wt. percent, inclusive.

12. The transparency of claim 11 wherein said first polymer concentration is about 3 to 4 wt. percent, inclusive.

13. The transparency of claim 1 wherein said coating solution further contains a surfactant effective in promoting adhesion of said coating to said surface.

14. The transparency of claim 13 wherein said surfactant is a mixture of anionic and nonionic surfactants and is present in said coating solution in a concentration in the range of about 0.05 to 2 wt. percent, inclusive.

15. The transparency of claim 1 wherein said coating solution further contains a sufficient quantity of hydrated alumina particles to impart pencil tooth to said coating.

16. The transparency of claim 15 wherein said alumina is present in said coating solution at a concentration of about 0.75 to 2 wt. percent, inclusive.

17. The transparency of claim 15 wherein said coating solution further contains wax-coated silica.

18. The transparency of claim 17 wherein said wax-coated silica is present in a concentration of about 0.1 to 0.3 wt. percent, inclusive.

19. The transparency of claim 1 wherein said substrate comprises a hydrophobic film and said surface is treated to be hydrophilic.

20. The transparency of claim 19 wherein said film comprises a polyester resin.

21. A method of forming a recording transparency comprising the steps of:

(a) providing a substantially transparent substrate having a hydrophilic surface; and,

(b) applying a substantially clear hydrophilic coating to said surface by coating said surface with an aqueous coating solution followed by evaporation of the water in said solution, said solution comprising one or more hydroxyethyl cellulose polymers dissolved in water, the concentration, average molecular weight, and molecular weight distribution of said polymer being selected such that said coating solution has a viscosity so as to be readily coatable on said surface and said coating provides a desired high rate of sorption and drying of indicia applied thereto.

22. The method of claim 21 wherein the concentration of said polymer in said coating solution is within the range of about 0.25 to 7 wt. percent, inclusive, and the viscosity of said solution is between about 100 and 4000 cps, inclusive, as measured at 25°C.

23. The method of claim 22 wherein said concentration is in the range of about 2 to 6 wt. percent, inclusive, and said viscosity is in the range of about 200 to about 2000 cps, inclusive.

24. The method of claim 22 wherein said solution comprises a mixture of hydroxyethyl cellulose polymers.

25. The method of claim 22 wherein a first said polymer is present in said coating solution and has a relatively low average molecular weight such that a 5 wt. percent aqueous reference solution thereof has a viscosity in the range of about 75 to 150 cps at 25°C.

26. The method of claim 25 wherein a second said polymer is present in said solution and has a relatively high average molecular weight such that a 2 wt. percent aqueous reference solution thereof has a viscosity in the range of about 4800 to 5600 cps at 25°C.

27. The method of claim 25 wherein a third said polymer is present in said solution and has a relatively high average molecular weight such that a 2 wt. percent aqueous reference solution thereof has a viscosity in the range of about 250 to 400 cps at 25°C.

28. The method of claim 26 wherein a third said polymer is present in said solution and has a relatively high average molecular weight such that a 2 wt. percent aqueous reference solution thereof has a viscosity in the range of about 250 to 400 cps at 25°C.

29. The method of claim 26, 27 or 28 wherein the concentration in said coating solution of said first polymer is in the range of about 0.25 to 7 wt. percent, inclusive, the concentration of said second polymer is up to about 1.5 wt. percent, and the concentration of said third polymer is in the range of 0 to 1 wt. percent, inclusive, provided however that the total concentration of said polymers is in the range of about 0.25 to 7 wt. percent, inclusive.

30. The method of claim 29 wherein said first polymer concentration is in the range of about 2 to 6 wt. percent, inclusive.

31. The method of claim 30 wherein said second polymer concentration is about 0.3 to 1.5 wt. percent, inclusive.

32. The method of claim 31 wherein said first polymer concentration is about 3 to 4 wt. percent, inclusive.

33. The method of claim 21 wherein said coating solution further contains a surfactant effective in promoting adhesion of said coating to said surface.

34. The method of claim 33 wherein said surfactant is a mixture of anionic and nonionic surfactants and is present in said coating solution in a concentration in the range of about 0.05 to 2 wt. percent, inclusive.

35. The method of claim 21 wherein said coating solution further contains a sufficient quantity of hydrated alumina particles to impart pencil tooth to said coating.

36. The method of claim 35 wherein said alumina is present in said coating solution at a concentration of about 0.75 to 2 wt. percent, inclusive.

37. The method of claim 35 wherein said coating solution further contains wax-coated silica.

38. The method of claim 37 wherein said wax-coated silica is present in a concentration of about 0.1 to 0.3 wt. percent, inclusive.

39. The method of claim 21 wherein said substrate comprises a hydrophobic resinous film and said surface is treated to be hydrophilic.

40. The method of claim 39 wherein said film comprises a polyester resin.

# INTERNATIONAL SEARCH REPORT

International Application No. PCT/US88/00589

**I. CLASSIFICATION OF SUBJECT MATTER** (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC (4): B41M 5/00, G01D 15/34, B32B 23/08, D06N 7/04
US 427/146, 256, 428/195, 206, 207, 211, 411.1

**II. FIELDS SEARCHED**

Minimum Documentation Searched [7]

| Classification System | Classification Symbols |
|---|---|
| US | 427/146, 256, 346/135.1, 428/195, 206, 207, 211, 411.1, 480 |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

**III. DOCUMENTS CONSIDERED TO BE RELEVANT** [9]

| Category * | Citation of Document, [11] with indication, where appropriate, of the relevant passages [12] | Relevant to Claim No. [13] |
|---|---|---|
| Y | US, A, 4,446,174 (MAEKAWA ET AL) PUBLISHED 01 MAY 1984, SEE ENTIRE DOCUMENT. | 1-40 |
| Y,P | US, A, 4,649,064 (JONES) PUBLISHED 10 MARCH 1987. SEE ENTIRE DOCUMENT. | 1-40 |

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**IV. CERTIFICATION**

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